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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 456

ANALYSIS OF SOAP

DETERMINATION OF FREE CAUSTIC ALKALI

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BRIEF HISTORY

The ISO Recommendation R 456, *Analysis of Soap. Determination of Free Caustic Alkali*, was drawn up by Technical Committee ISO/TC 91, *Surface Active Agents*, the Secretariat of which is held by the Association Française de Normalisation (AFNOR).

Work on this question by the Technical Committee began in 1961 and led, in 1962, to the adoption of a Draft ISO Recommendation.

In June 1963, this Draft ISO Recommendation (No. 584) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies:

Argentina	Hungary	Poland
Austria	Italy	Portugal
Canada	Japan	Romania
Chile	Korea, Rep. of	Spain
Colombia	Morocco	Switzerland
Czechoslovakia	Netherlands	United Kingdom
France	New Zealand	Yugoslavia
Germany	Norway	

One Member Body opposed the approval of the Draft: India.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in November 1965, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 456 November 1965

ANALYSIS OF SOAP

DETERMINATION OF FREE CAUSTIC ALKALI

INTRODUCTION

As soaps usually contain a small amount of unsaponified neutral fat, there is no perfect procedure for determining free caustic alkali because, when the sample of soap is dissolved, a neutral fat is more or less saponified by any free caustic alkali that is present.

Both methods described in this ISO Recommendation are therefore of a conventional nature.

Conventionally, free caustic alkali is expressed as

sodium hydroxide (NaOH) for sodium soaps and

potassium hydroxide (KOH) for potassium soaps.

1. SCOPE

The purpose of this ISO Recommendation is to specify two methods of determining free caustic alkali in commercial soaps, excluding compounded products:

Method A, ethanol method,

Method B, barium chloride method.

1.1 Choice of method

Method A (ethanol method) should be applied only to sodium soaps of ordinary quality, as the presence of certain additives brings in sources of error. It is not applicable to potassium soaps, because of the solubility of potassium carbonate in ethanol.

Method B (barium chloride method) should be applied to all soft potassium soaps or mixed sodium and potassium soaps. The application of this method to sodium soaps of ordinary quality, which usually do not contain sufficient quantities of free caustic alkali to be determined by this method, is not recommended.

2. TERMINOLOGY

It is understood that *free caustic alkali in a soft soap* is the quantity of hydroxyl ion, reported as potassium hydroxide (KOH), which is found in solution after precipitation with barium chloride under the operating conditions described.

3. METHOD A

(Ethanol method)

3.1 Principle

The soap is dissolved in neutralized ethanol, and the free caustic alkali is titrated with an ethanolic solution of hydrochloric acid.

3.2 Reagents

3.2.1 *Ethanol*, absolute.

3.2.2 *Potassium hydroxide*, ethanolic solution, approximately 0.1 N.

3.2.3 *Hydrochloric acid*, ethanolic solution, approximately 0.1 N.

3.2.4 *Phenolphthalein solution*, 1 g in 100 ml of 95 per cent by volume ethanol.

3.3 Apparatus

Ordinary laboratory apparatus, and in particular

(*a*) Flask of approximately 400 ml capacity, that can be fitted to a reflux condenser ;

(*b*) Reflux condenser ;

(*c*) Analytical balance.

3.4 Procedure

3.4.1 *Test portion.* Weigh approximately 5 g of soap to an accuracy of 0.01 g.

3.4.2 *Determination.* Pour 200 ml of ethanol (3.2.1) into the flask. Connect to the reflux condenser. Bring to a gentle boil and keep at the boil for 5 min in order to remove carbon dioxide. Remove from the condenser and allow to cool to about 70 °C. Add 4 drops of phenolphthalein indicator (3.2.4). Neutralize exactly with the ethanolic solution of potassium hydroxide (3.2.2), until the indicator just turns pink.

Place the test portion in the flask containing the neutralized ethanol. Connect the flask to the reflux condenser and boil gently until the soap has completely dissolved. Cool to about 70°C. Titrate with the ethanolic solution of hydrochloric acid (3.2.3) until the colour is just perceptibly pink, identical with that obtained when the ethanol was neutralized.

3.5 Expression of results

3.5.1 *Method of calculation and formula*

If

E is the mass in grammes of the test portion,

n is the number of millilitres of ethanolic hydrochloric acid solution (3.2.3) used,

T is the normality of the ethanolic hydrochloric acid solution (3.2.3) used,

the percentage of free caustic alkali in the soap, expressed as sodium hydroxide (NaOH), is

$$0.040 \times n \times T \times \frac{100}{E}$$

The free caustic alkali content can be also expressed in milliequivalents per kilogramme by means of the following formula:

$$\frac{n \times T}{E}$$

3.5.2 *Reproducibility.* ± 0.02 absolute.